# FY91 Semi-Aqueous Work

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**ABSTRACT:** This document consists of information memos and other items that record the efforts expended in FY91 to evaluate the semi-aqueous cleaning method. It must be supplemented by other documents available from the contact person listed, or as noted.

**APPLICABILITY:** This document is for informational use only.

**STATUS:** APPROVED 30-Aug-1991; use VTX SMC for current status.

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# 1 INTRODUCTION

Studies of component compatibility, relative need for treatment of waste materials, costs in use, relative cleaning efficacy, safety and health issues, and various related documents from suppliers and users are among the items included in this document.

The names of the authors of the included documents constitute a reference list of contacts both internal to Digital™ and outside.

## 1.1 BACKGROUND HISTORY

With the support of Tom Newbold, Senior Program Manager, managing the USSM CFC (chlorofluorocarbon) Elimination Program, Anne Reynolds and Paul Thibodeau worked within the CFC Alternatives space to better understand and characterize the various module manufacturing cleaning alternatives. An early literature search, supplier visits, and trade show surveys indicated that much valuable work and information was available. The work of the IPC Ad Hoc Solvents Committee was especially timely and valuable. This work allowed them to concentrate on a the few most likely viable alternatives; those that clearly cleaned as well as, or better than, CFC-113.

## 1.2 WHEN TO USE THIS DOCUMENT

As interest increases in the areas of product cleaning, and saponified aqueous cleaning is realized to be environmentally compromising, these documents will be of great interest. Items and issues that might be considered indicators include the following.

- Imminent purchase of a cleaning system
- Increase of cost of water and saponifier
- Increases in government regulation of saponifier, its constituents, and its by-products
- Finer technology that requires increased cleaner penetration
- Decrease of water availability
- Increased attractiveness of closed loop recyclability
- Toxicity of base chemicals (such as glycol ether, terpene, and so forth)

## 1.3 EXPECTED USERS OF THIS DOCUMENT

Decision makers, either financial or technical, will find various portions of this document helpful.

Site Manufacturing Engineers in charge of the cleaning process will use this document to avoid repeating earlier efforts and to obtain a history of that which has been done to date.

Development Manufacturing Engineers will use this document as a reference to that which has been done, as a guide to contacts, and to avoid repeating earlier efforts.

Environmental Health and Safety (EH&S) engineers will use this document while evaluating toxic potentials, health and safety issues, compliance with local, regional, and national regulations.

# 2 CLEANING

## 2.1 EXTERNAL REVIEW DRAFT AQUEOUS AND TERPENE CLEANING INTERIM REPORT— NOV. 15, 1990

## 2.2 MANUAL OF PRACTICES TO REDUCE AND ELIMINATE CFC-113 USE IN THE ELECTRONIC INDUSTRY

## 2.3 SEMI-AQUEOUS CLEANING—A BUSINESS NICHE WITHIN DIGITAL

## 2.4 EUROPE: CFC UPDATE, OCTOBER 22, 1991

## 2.5 USSM CFC DISTRIBUTION LIST

## 2.6 CFC UPDATE

## 2.7 COMPONENT CONCERNS ON USING TERPENE CLEANING SOLUTION

## 2.8 EC-7R CONDUCTIVITY

## 2.9 EC-7R CONDUCTIVITY... ANOTHER??

## 2.10 EC-7M AND EC-7R TERPENE AS CLEANERS

## 2.11 SOLVENT TO COMPONENT COMPATIBILITY MATRIX—A PROPOSAL

## 2.12 EXPERIMENTAL RESULTS OF BOACT EC-7M OPEN VAT CLEANING STUDY

## 2.13 FEASIBILITY REPORT ALTERNATIVE CLEANING (FOCUS ON SEMI-AQUEOUS)

## 2.14 MATERIAL SAFETY DATA SHEET (MSDS)

## 2.15 AXAREL 52 MSDS

## 2.16 OTHER DOCUMENTS

# 3 ENVIRONMENTAL HEALTH AND SAFETY

## 3.1 NCMS ENVIRONMENTAL ISSUES IN MANUFACTURING - A CONTACT LIST

## 3.2 TERPENE HAZARD ASSESSMENT—NOVEMBER 5, 1990

## 3.3 BIOACT EC-7R MSDS

## 3.4 KCD-9477 MSDS

## 3.5 KYZEN IONOX MC MSDS

## 3.6 CONCERNS ON USING TERPENE CLEANING SOLUTION

## 3.7 HEALTH AND SAFETY EVALUATION FOR USE OF EC-7, A TERPENE PRODUCT

## 3.8 EH&S EVALUATION OF EC-7

## 3.9 PRJ APPROVAL/EC-7 INDUSTRIAL HYGIENE AND TOXICOLOGY

## 3.10 OTHER DOCUMENTS

# 4 WASTE TREATMENT

## 4.1 OTHER DOCUMENTS

# 5 EQUIPMENT

## 5.1 A COMPARISON OF AQUEOUS AND SEMI-AQUEOUS CLEANING EFFLUENT

**NOTE**

**Contact Anne Reynolds or Paul Thibodeau (See VTX ELF for location information) for additional documents included in this report.**

## A COMPARISON OF AQUEOUS AND SEMI-AQUEOUS CLEANING EFFLUENT

**Anne Reynolds, APO Environmental Engineering, and Paul Thibodeau, TME/MPE**

**Executive Summary** Two semi-aqueous materials were compared with a saponified aqueous process to assess differences in the characteristics of effluent produced using a batch cleaning system. Two types of test vehicles were used and cleanliness was evaluated using visual inspection and ionic contamination by Ionograph. The test vehicles were also cleaned with a 1,1,1 trichloroethane-based cleaner and a Freon 113 cleaner. (See attached diagram.)

**Test Results**
*COD levels* - CODs from the saponified aqueous cleaner were of a much higher magnitude than either of the semi-aqueous cleaners. Of the semi-aqueous materials, EC-7R compared favorably to Axarel 38. Contamination from the saponified aqueous rinse water alone did approach the levels of the semi-aqueous processes. *Lead levels* - Only the lead levels in the saponified aqueous process approached typical discharge limits for lead.

*Neutraliazation requirements* - Results showed that the saponified cleaner required the largest amount of acid. The semi-aqueous cleaners required far less with EC-7R requiring the least.

**Conclusions and Recommendations**
The study primarily showed the relative amounts of contamination that would need to be removed in order to reuse process water. EC-7R, because it readily separates from water clearly offers the best opportunity for water recycling with the least amount of work for contaminant removal. Axarel 38, most likely because it is more miscible, did not compare favorably with EC-7R in any contaminant. The rinse water from saponified aqueous cleaning appears to offer some possibility of allowing for water recycling but it appears to offer the least. Also the wash water in this case would require treatment either on or off site. Although both semi-aqueous materials will probably require off-site treatment, the volumes generated would be much lower than the volumes of saponified wash water generated, although this study did not provide data on this. Graph 6 (and expanded version 6A) shows the relative ratios of contaminants for all parameters. Now, all cleaning methods are continuing to improve. A phase-separable version of Axarel has been introduced by DuPont. Formulators of saponifier are working on longer lasting mixtures to increase re-use of wash water. More data concerning other issues including workplace health and safety, component compatibility and cleaning ability continues to become available. These developments should continue to be investigated. When aqueous processes are required, water reuse will prevent air and water contamination, will conserve energy and resources and may reduce the regulatory burden. At the present time EC-7R offers the easiest path to water reuse.

### A Comparison of Aqueous and Semi-Aqueous Cleaning Effluent

**Introduction**
Many, but not all, of Digital's manufacturing plants, have used aqueous module cleaning methods instead of using Freon or other chlorinated solvents. With a greater focus on semi-aqueous methods in the industry, as well as an awareness of some of the disadvantages of aqueous cleaning, a relative comparison was warranted. Digital is fortunate to have available ECD (1) equipment designed for small batch cleaning. There is much data available about the relative cleaning ability of these various methods. One of

the key differences in cleaning is in the waste by products produced, so our study focused on this issue. Due to limited funding and resources, the experiment was designed to be as simple as possible, using easily measured parameters. The goal was to determine whether further study of cleaning alternatives is necessary instead of attempting to find definitive answers at this time.

The batch cleaning system was the basis for the process and two types of available test vehicles were cleaned using different chemicals at room temperature. Various parameters of the effluent were measured. Test vehicles were also cleaned using a 1,1,1-trichloroethane formulation in a degreaser and in a Freon 113 mixture. These processes do not produce any aqueous effluent. The test vehicles were inspected visually and took ionograph readings to verify relative cleaning ability. (See attached diagram.)

**Method**

1. Test vehicle coupons

   ENVIRONMENTAL - Small coupons (4 X 4.5 in) with sites for six 25 mil pitch (1 in. sq.) surface mount components were used. The boards were marked and initially cleaned using Alconox and water, a deionized water (DI) rinse and an alcohol rinse. The dry boards were then weighed, pasted on one side and weighed again. All of the boards were weighed to compare uniformity. Two devices were randomly placed on the boards and they were reflowed in an oven. Time constraints prevented good control of this reflow process and results were not uniform. Three boards were processed but not cleaned to use as controls. The boards were then divided into batches of ten boards. One batch was cleaned using Freon 113 (2). one batch was cleaned in a degreaser using Prelete (3)(A formulation of 1,1,1-trichloroethane.) Three batches were cleaned using a 2-aminoethanol, 2-butoxyethanol based saponifier similar to one used at many Digital sites. Three batches were also cleaned using Axarel 38 (4) and three batches were cleaned using EC-7R (5). Both of these are semi-aqueous cleaners.

   The ECD batch system involves two dishwasher-like machines. Due to the combustible nature of some of the semi-aqueous solvents the model 6307 sprays the modules with the semi-aqueous material in a nitrogen atmosphere which prevents ignition. The material then drains into a reservoir (usually a 5gal metal drum) and the material is reused. The machine monitors the specific gravity of the semi-aqueous liquid as a control for the of the amount of dissolved flux contained in the liquid.

   After the rack has drained it is moved to the 6300 machine where it is rinsed. Each rinse cycle consists of 2.8 gal of water. The first cycle is considered a prewash and the second cycle is a wash and saponifier can be added at this point. Times for these two cycles can be adjusted. Subsequent rinses are uniform. The machine monitors water temperature and resistivity and the number of rinses can be set or it will continue rinse cycles until a set resistivity is reached. The machine requires an additional water heater, a drain, and the use of what ever water treatment is desired. Our machine was plumbed to allow 18 megohm water or 0.1 megohm DI water. For cleaning the test vehicle, we used DI water.

   When using a saponifier, only the 6300 is used. It is equipped with two pumps, one for saponifier and one for de-foamer. Concentration of saponifier can be set. For all cycles in this comparison, the prewash was set at 1 min, the wash at 7 min and 9 rinse cycles were completed. A 7% saponifier solution was used as the wash cycle of the saponified aqueous batches.

Each prewash and wash were sampled separately. Two rinse cycles were collected together. All samples were analyzed for pH, most for chemical oxygen demand (COD, a measure of organic contamination) and some for both total and dissolved lead and copper. There was very little copper so these results are not included. Resistivity measurements for each cycle were recorded by the machine and the position of each coupon in the rack was recorded for possible correlation later.

The samples were then composited into a sample representative of the total volume of effluent and this was titrated with sulfuric acid to obtain a measure of the volume of acid required to neutralize each waste stream.

CLEANLINESS - All of the processed boards were visually inspected using the following scoring method:

0 = Clean solder joints; no visible residue
2 = Solder joints with some residue present
5 = Visible flux or large amounts of residue

Ionic contamination on some of the boards was measured using an Ionograph 500.

2. Product boards

Two versions of a product board were obtained and divided into 3 batches of 7 boards. Each of these was processed similarly to the method used above, however these were reflowed in a more controlled process in an IR inline reflow oven. In this cleaning process 18 megohm water was used and 3 extra boards were cleaned using the water alone.

**Test Results**

**COD levels:** The COD is a measure of the amount of organic contamination in water and waste water. There are a variety of treatment methods for COD which may range from carbon absorption for small quantities to chemical or biological oxidation. The need for treatment will depend on local conditions for treatment of industrial effluent and whether the contributed flow is a significant contribution to the discharge point and on the local capacity for organic treatment.

Graph 1 (Graph 1A gives an expanded view of the semi-aqueous processes) shows the average COD levels in effluent including and excluding the wash cycle. The data for the total COD was calculated by determinining the cumulative total COD for the whole process divided by the total volume of water used. The rinse data was calculated by subtracting the highest level COD and the volume of that rinse from the total volume of water. This was an attempt to simulate conditions for recycling the wash cycle for saponified aqueous cleaning or using an emulsion stage and decant for semi-aqueous processing. The data for the test vehicle batches were averaged.

CODs from the saponified aqueous cleaning were of a much higher magnitude than those of either semi-aqueous cleaner as expected. Axarel 38 was consistently higher in COD than EC-7R. CODs from the product boards were lower than CODs for the test vehicles even though the total surface area of the product boards was almost 4 times higher than the test vehicles. This was unexpected and may have been due to the fact that the boards were not rinsed as quickly as the test vehicles and a significant amount of material evaporated. The COD in a semi-aqueous process should be related to the dragout on the boards and racks and although this is related to the total surface area, it will also depend on the amount of time allowed for draining and evaporation. The CODs from the saponified aqueous cleaning would depend more on the initial concentration of saponifier used, but the contamination

in the rinse water would reflect the dragout of wash solution. The results reflected this as the CODs for the product boards rinse water are more proportional to the area and the solder paste weight.

**Lead levels:**

Lead is a very significant toxin and is usually chemically treated or removed by ion exchange. Although it is present in small amounts in modules processing treatment, limits for treatment are becoming increasingly more stringent, frequently approaching detection limits.

Samples were analyzed for lead by ICP on filtered and unfiltered samples. The numbers used for the graph were the maximum lead levels found during the process, usually from the wash cycle. As expected, higher levels were found on the product boards than on the test vehicles for EC-7R and for the saponifier, but lower levels were encountered with Axarel 38. Only the product boards cleaned with saponifier approached typical discharge limits for lead. (Refer to Graph 2.)

**Neutralization requirements**

PH adjustment is generally accomplished by acid or caustic additions, but requires the storage and handling of corrosive materials and the treated waste water will contain increased levels of salt as a result of the neutralization.

A composite sample representative of the total rinse was prepared for each chemistry. These were titrated to determine the total quantity of acid required to neutralize the entire batch effluent. Results showed that the saponified cleaner required the highest amounts of acid. The semi-aqueous cleaners required far less with EC-7R requiring the least. (Refer to Graph 3.)

**Cleaning ability**

There are various methods of measuring cleaning ability. Since there is a large amount of data available about relative cleaning abilities our effort in this area consisted of verifying reported results with our own test vehicle.

We had initially hoped our study would provide more information regarding cleaning ability. The variation in results on the test vehicles is most likely due to not being able to closely control reflow temperatures and exposures. Reflow of the product boards more closely controlled.

Visual average results (30 test vehicles for each aqueous or saponified aqueous methods, 10 for Freon and 1,1,1) showed close correlation of both semi-aqueous and saponified aqueous with solvent cleaning. (See graph 4.) Visual scores for the product boards showed Axarel as slightly better than saponifier and both better than EC-7R. (See graph 5.)

Ionograph reading were extremely low for all boards probably reflecting low surface area and the fact that the boards were relatively unpopulated. Graphs of this data are not included as it did not prove to be very meaningful.

## Conclusions and Recommendations

The study primarily showed the relative amounts of contamination that would need to be removed in order to reuse process water. EC-7R, because it readily separates from water clearly offers the best opportunity for water recycling with the least amount of work for contaminant removal. Axarel 38, most likely because it is more miscible did not compare favorably with EC-7R in any contaminant. The rinse water from saponified aqueous cleaning appears to offer some possibility of allowing for water recycling but it appears to offer the least. Also the wash water in this case would require treatment either on or off site. Although both semi-aqueous materials will probably require off-site treatment, the volumes generated would be much lower than the volumes of saponified wash water generated, although this study did not provide data on this. Graph 6 (and expanded version 6A) shows the relative ratios of contaminants for all parameters. At this time, all cleaning methods are continuing to improve. A phase-separable version of Axarel has been introduced by DuPont. Formulators of saponifier are working on longer lasting mixtures to increase re-use of wash water. More data concerning other issues including workplace health and safety, component compatibility and cleaning ability continues to become available. These developments should continue to be investigated. When aqueous processes are required, water reuse will prevent air and water contamination, will conserve energy and resources and may reduce the regulatory burden. At the present time EC-7R offers the easiest path to water reuse.

## Acknowledgements

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1. ECD is a trademark of Electronic Controls Design, Inc., Milwaukie, OR 97222-8825.
2. Freon is a trademark for chloroflorocarbon products manufactured by DuPont Chemicals, Wilmington, DE 19898.
3. Prelete is a trademark for Defluxer solvent manufactured by Dow Chemical U.S.A., Midland, MI 48674.
4. Axarel 38 is a trademark for a hydrocarbon semi-aqueous cleaner manufactured by DuPont Chemicals, Inc., Wilmington, DE 19898. See attached MSDS.
5. Bio-Act EC-7R is a trademark for a terpene based semi-aquoeus cleaner manufactured by Petroferm, Inc., Fernandina Beach, FL 32034. See attached MSDS and data sheet.
6. Alconox is a trademark for a detergent manufactured by Alconox, Inc., New York, NY 10003.

## 5.2 CONTACTS FOR WASTE TREATMENT

## 5.3 EC-7R SIMONS WASTE TREATMENT IN IOMEGA AT SALT LAKE CITY

## 5.4 MOTOROLA SEMI-AQUEOUS QUESTIONS AND AGENDA ITEMS

## 5.5 WASTE TREATMENT AND RECYCLING À LA EC-7R

## 5.6 OTHER DOCUMENTS

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